EXHIBIT I

Chemistry Unit
Validation of Analytical Procedures

1 Purpose

This document supplements the *FBI Laboratory Practices for Developing Technical Procedures, FBI Laboratory Practices for Validating Technical Procedures, FBI Laboratory Practices for Validating Chemical Procedures*, and the *FBI Laboratory Quality Assurance Manual* for the development and validation of new analytical procedures in the Chemistry Unit (CU).

2 Scope

This document applies to all new analytical procedures developed and validated in the CU. The development and validation of an analytical procedure is referred to as a validation study in the CU. The performance characteristics that are evaluated during a validation study will be based on the scope of the analytical procedure. The validation study must be completed, reviewed, and approved prior to the procedure's first use in casework, except as noted within this procedure. Review and approval of a validation study includes the review and approval of the associated development (when applicable) and validation study records.

3 Responsibilities

3.1 The Individual/Group performing the validation study will:

- Develop, document, and ensure approval of a validation plan utilizing the applicable CU *Validation Plan* form (Appendices A-C). Retain the approved CU *Validation Plan* form.
- Document and retain the results of the validation study. This documentation may include development study data, validation data, instrument optimization charts, relevant literature references, etc.
- Ensure approval of the validation study utilizing the *Validation of Chemical Procedures Review Form* (7-267) or the CU *Validation Plan and Review- Physical Properties Only* form (Appendix C), as applicable.
- All documentation related to validation studies conducted within the CU will be maintained within binders, in electronic format, or within case notes, as appropriate. Validation study binders will be stored within the CU's file cabinets and/or bookshelves.

3.2 The applicable Technical Reviewer(s) will:

- Review and approve validation records. Document these reviews on the 7-267 or CU *Validation Plan and Review- Physical Properties Only* form (when applicable).

3.3 The applicable Subunit Manager will:

- Approve validation plans by signing the applicable CU *Validation Plan* form.
- Approve validation studies by signing the "Technical Leader Approval" line on the 7-267 (when applicable) or the CU *Validation Plan and Review- Physical Properties Only* form (when applicable).

3.4 The CU Chief will:

- Approve validation plans by signing the CU *Validation Plan* form.
- Approve validation studies by signing the 7-267 or the CU *Validation Plan and Review- Physical Properties Only* form (when applicable).

4 Practices

4.1 Validation Studies

Validation studies of chemical procedures in the CU will be performed following the requirements outlined in the *FBI Laboratory Practices for Validating Chemical Procedures*. These requirements may be adjusted based on the scope of the procedure and professional judgment (e.g., safety considerations, differences in sample matrices, availability of reference materials).

Validation studies of applicable, non-chemical procedures (i.e., physical property measurements) in the CU will be limited to the characteristic listed in section 4.1.1 of this document.

Validation studies for casework involving the analysis of unknowns will be conducted as detailed in section 4.1.2 of this document.

4.1.1 Performance Characteristic for Measurement of a Physical Property

- Accuracy

4.1.1.1 Accuracy

Accuracy is the closeness of an analytical result to its true value and is affected by systematic error (bias) and random error (precision). The accuracy of a physical property measurement can be determined by comparison of that measurement result with the true value. At a minimum, ten measurement replicates of a reference material with a known physical property value are made. The accuracy is calculated as the percent difference of the average measured value from the known value. In most instances, the preferred accuracy is ±15% or less, but larger values may be unavoidable and are acceptable if accompanied by proper justification.

4.1.2 Performance Characteristics for the Analysis of Unknown Component(s)

Due to the nature of unknown component analysis, the validation may be conducted either at the time of analysis or immediately following. Generally, the only performance characteristic that needs to be validated is selectivity.[1]

4.2 Document, Review, and Maintain Validation Study Results

Upon completion of the validation study, the CU Chief or designee will assign appropriate personnel as technical reviewer(s) of the validation study results. If the CU Chief is qualified to do so, he/she may perform the technical review. The technical review must take place before the procedure is placed into use.[2] The technical reviewer(s) will complete the 7-267 or the CU *Validation Plan and Review- Physical Properties Only* form, whichever is applicable. After the technical review is complete, the CU Chief and appropriate Subunit Manager (as applicable) will review the validation records and document their approval on the 7-267 or the CU *Validation Plan and Review- Physical Properties Only* form. The completed review form will be maintained with the validation study data. Once a new analytical procedure has final approval by the CU Chief, and before it is used in casework, the procedure will be formally written and reviewed following the appropriate Laboratory Division and CU practices.

In extreme situations (e.g., court mandates) when a validated procedure must be used prior to being formally written and through all reviews, it is permissible to use the validated procedure for casework provided that the same steps for sample preparation and instrumental parameters used during the validation are also used for the analysis and there is clear, written documentation of the steps that were taken to generate the results. In these instances, at a minimum, the validation data will be technically reviewed by another qualified Examiner in the category of testing prior to using the validated procedure for casework. This will be treated as a minor or major deviation, as appropriate, according to the *FBI Laboratory Practices for Authorizing Deviations*.

Validation study records will be maintained within the CU's appropriate validation file cabinets, bookshelves, in electronic format, or, with respect to case-specific validation, in the related case notes.

When a validation study has been performed for what is most likely to be a one-time analysis, a validated procedure can be applied in casework without the issuance of an official standard operating procedure. In these instances, the following criteria will be met:

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[1] In this scenario, the requirement of completing a 7-267 will be waived.

[2] The exception to this rule will be when validating the analysis of an unknown component. In these cases, the 7-267 will not be required and the review of the validation will occur as part of the Laboratory Report technical review.

- A validation plan will be created and reviewed using the appropriate CU *Validation Plan* form, and approved prior to commencing validation.
- Step-by-step instructions for the analysis and a summary of the validation performed will be prepared and retained with the validation records.
- The validation records will be technically reviewed and approved by the CU Chief and appropriate Subunit Manager, if applicable. This will be documented on the 7-267 or on the CU *Validation Plan and Review-Physical Properties Only* form, whichever is applicable.
- A copy of the applicable review form and a copy of the step-by-step instructions will be retained in the case notes for the affected case.
- The validation records will be stored in a central location to include the CU's appropriate validation file cabinets or bookshelves.
- If and when the procedure is performed again, a standard operating procedure will be written. Required reviews and approvals will be obtained before issuance of the new procedure.

5 Competency Testing on Newly Validated Analytical Procedures

Examiners and Chemists must successfully complete a competency test on a newly validated analytical procedure prior to applying the procedure to casework. This test will demonstrate that the Examiner or Chemist can accurately perform the procedure. For personnel that were involved in the validation process, the applicable Subunit Manager or CU Chief may approve the validation work to serve as demonstration of competency. The successful completion of a competency test, or the approval to use validation work as a substitute for a competency test, will be documented in the employee's Training and Qualification Records binder.

6 Minor Deviations to Previously Validated Procedures

Minor deviations to standard operating procedures in the CU will be considered for approval by the requestor's Subunit Manager. If the requestor is a Subunit Manager, the minor deviation request will be considered for approval by the CU Chief. If the CU Chief is not qualified in the procedure in question, the CU Chief will consult with an Examiner that is qualified in the procedure and both the CU Chief and the Examiner will document their approval of the minor deviation.

6.1 Subunit Logs of Minor Deviations

To maintain consistency when other CU personnel are faced with the same or similar analyses, all minor deviations to standard operating procedures will be recorded in Subunit logs maintained by the Subunit Manager. At a minimum, each log will include the FBI Laboratory number associated with the minor deviation, the analyte(s) that were targeted, the date of the minor deviation, the personnel that performed the minor deviation, the personnel that approved the minor deviation, the

name of the procedure affected, and any additional relevant information. This will allow for review of the minor deviation if the documentation resides in a specific case file.

7 References

FBI Laboratory Practices for Validating Chemical Procedures, FBI Laboratory Operations Manual.

FBI Laboratory Practices for Validating Technical Procedures, FBI Laboratory Operations Manual.

FBI Laboratory Practices for Developing Technical Procedures, FBI Laboratory Operations Manual.

LeBeau, M. et al. "Validation Guidelines for Laboratories Performing Forensic Analysis of Chemical Terrorism", *Forensic Science Communications,* 7(2), April 2005.

Peters, F.T. "Bioanalytical Method Validation and its Implications in Forensic and Clinical Toxicology - A Review", *Accred. Qual. Assur.* 7, 2002, 441-449.

Peters, F.T., Drummer, O.H., and Musshoff, F. "Validation of New Methods", *Forensic Science International*, 165(2-3), 2007, 216-224.

Rev. #	Issue Date:	History:
6	11/06/12	Removed "LOM" throughout when referencing *FBI Laboratory Practices*. Simplified section 1 to clarify that this is a supplemental document. Defined validation study in section 2 to include development and validation, removed "development" throughout (where applicable). Added review and approval of a validation study includes development study records (when applicable) in section 2. Edited sections 3.1-3.4 for clarity. Added electronic format as storage option in sections 3.1 and 4.2. Replaced "*Validation of Chemical Procedures Review Form* (7-267)" with "7-267" throughout (after defining in section 3.1). Changed 4.1 heading and edited section for clarity. Edited content of section 4.1.2.2 to apply to physical property measurements (rather than chemical properties). Changed section 4.1.3 and footnote 2 from "identification of unknown component(s)" to "analysis of unknown component(s)". Edited section 4.2 for clarity. Added competency demonstration via validation study work and documentation requirements to section 5. Changed "Examiner/Chemist" to "person" in section 6. Deleted Appendices A, B, & C and references to them in sections 3.1 and 4.2 as they are available on the FBI Intranet.
7	04/18/13	Defined Chemistry Unit as "CU" in section 1 and used throughout document. Replaced "Unit Chief" with "CU Chief" throughout. Appendices A-C were revised and added back to document. Appendix C was revised to include documentation of a validation plan, and the title was appropriately changed. Added references to the appendices in section 3.1. Changed title of section 4.1 (was "Validation of Chemical Procedures"). Renumbered section 4.1 to fix error (previous version was missing section 4.1.1). Changed section 4.1.1 bullet from "Accuracy (Bias)" to "Accuracy". Merged previous sections 4.1.2.1 and 4.1.2.2 (now section 4.1.1.1) and changed "bias" to "accuracy" where applicable. Added section 6 covering minor deviation approval. Previous section 6 became section 6.1 and "modification" changed to "minor deviation" throughout section 6.1. Changed "person" to "personnel" in section 6.1.

Approval

Chemistry Unit Chief: Michael A. Smith Date: 04/17/2013

Michael A. Smith

QA Approval

Quality Manager: Catherine E. Theisen Date: 04/17/2013

Catherine E. Theisen

Issuance

Unit QA Manager: Jason D. Brewer Date: 04/17/2013

Jason D. Brewer

Appendix A: Validation Plan for Qualitative Procedures

(04-18-13)

VALIDATION PLAN Scope-*Qualitative* Procedure

CHEMISTRY UNIT // FBI Laboratory

Scope: Click here to enter text.

Matrix(ces): Click here to enter text.
Analyte(s): Click here to enter text.
Instrumentation: Click here to enter text.
Analytical Method(s): Click here to enter text.
Sample Preparation: Click here to enter text.

Acceptable Limits

☐ Limit of Detection: Click here to enter text.
☐ Processed Sample Stability: Click here to enter text.
☐ Selectivity: Click here to enter text.
☐ Ionization Suppression/Enhancement: Click here to enter text.

Other Information: Click here to enter text.

Lead Scientist: _____ Date: _____

Subunit Manager Approval: _____ Date: _____

Unit Chief Approval: _____ Date: _____

Appendix B: Validation Plan for Quantitative Procedures

(04-18-13)

VALIDATION PLAN Scope-*Quantitative* Procedure

CHEMISTRY UNIT // FBI Laboratory

Scope: Click here to enter text.

Matrix(ces): Click here to enter text.
Analyte(s): Click here to enter text.
Instrumentation: Click here to enter text.
Analytical Method(s): Click here to enter text.
Sample Preparation: Click here to enter text.

Acceptable Limits

- ☐ Accuracy: Click here to enter text.
- ☐ Calibration Model: Click here to enter text.
- ☐ Carryover: Click here to enter text.
- ☐ Ionization Suppression/Enhancement: Click here to enter text.
- ☐ Limit of Detection: Click here to enter text.
- ☐ Limit of Quantitation: Click here to enter text.
- ☐ Precision: Click here to enter text.
- ☐ Processed Sample Stability: Click here to enter text.
- ☐ Recovery: Click here to enter text.
- ☐ Selectivity: Click here to enter text.

Other Information: Click here to enter text.

Lead Scientist: _____ Date: _____

Subunit Manager Approval: _____ Date: _____

Unit Chief Approval: _____ Date: _____

Appendix C: Validation Plan and Review for Physical Property Measurements

(04-18-13)

VALIDATION PLAN AND REVIEW

Scope-Physical Properties Only

CHEMISTRY UNIT // FBI Laboratory

Procedure Name: Click here to enter text.

Scope: Click here to enter text.

Intended Use: ☐ Measurement of Physical Property

Validation Plan: Click here to enter text.

Lead Scientist: _____ Date: _____

Subunit Manager Approval: _____ Date: _____

Unit Chief Approval: _____ Date: _____

Validation Review:

Performance Characteristic	Date	Lead Scientist	Reviewer
☐ Accuracy	_____	_____	_____

This method has been appropriately validated for its intended use (as specified above). The method is considered fit for the above use.

Unit Chief Approval: _____ Date: _____

Subunit Manager Approval: _____ Date: _____